

UCRL-JC-132939

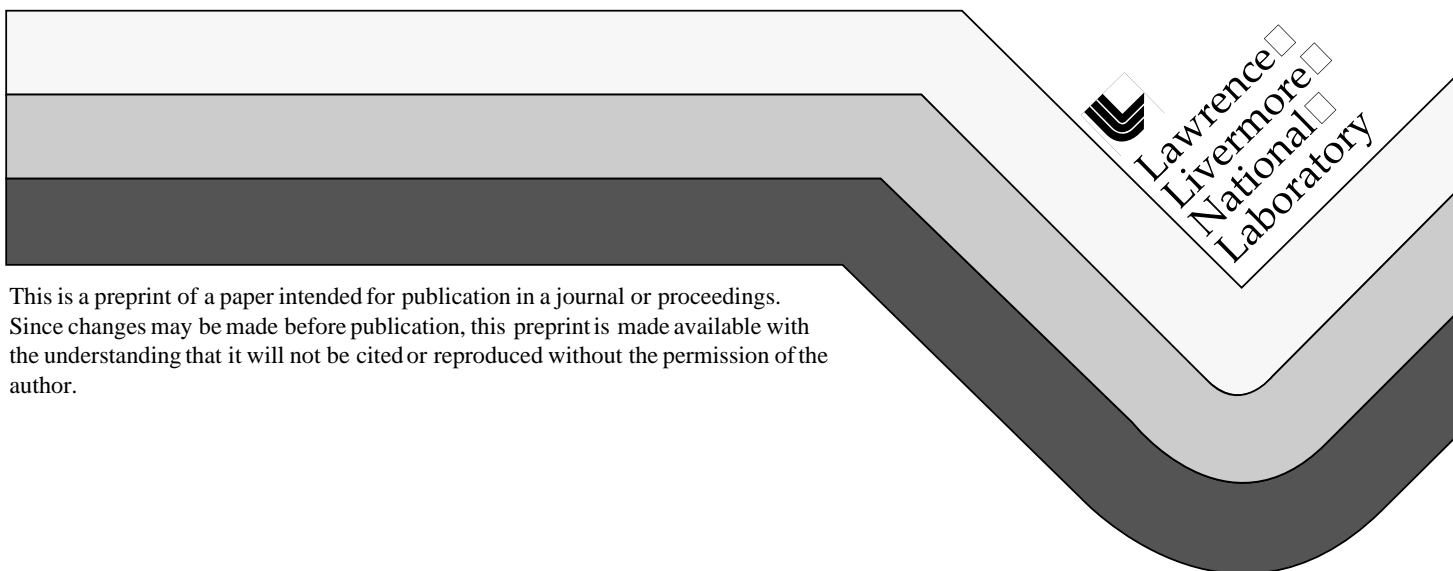
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This paper was prepared for submittal to the
44th Annual Meeting of the International Symposium on
Optical Science, Engineering, and Instrumentation
Denver, Colorado
July 18-23, 1999

July 1999



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Optical Cleanliness Specifications and Cleanliness Verification

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ABSTRACT

Optical cleanliness is important to NIF because it results in beam obscuration and scatter losses which occur in the front-end (containing over 20,000 small optics) and the large-aperture portions of the laser (containing ^a 7,300 optics in 192 beamlines). The level of particulate cleanliness necessary for NIF, is similar to the scatter loss due to surface roughness. That is, the scatter loss should not exceed $\leq 2.5 \times 10^{-5}$ per surface.

Establishing requirements for optical and structural surface cleanliness needs consideration of both particulate and organic thin-film cleanliness. Both forms of cleanliness may be specified using guidelines specified in Military Standard 1246C and are referred to as cleanliness Levels. This Military Standard is described briefly and displayed in tables and charts. The presence of organic thin-films on structural surfaces is of particular concern if the contaminated surface is near solgel coated optics (solgel coatings provide an antireflection (AR) quality); or the optic is in a vacuum. In a vacuum, organic contaminant molecules have a much high probability of transporting from their source to a solgel-coated optic and thereby result in the rapid change in the transmission of the antireflection coating.

Optical surface cleanliness can be rapidly degraded if a clean optic is exposed to any atmosphere containing an aerosol of small particles. The use of cleanrooms, as described in Federal Standard 209C, minimizes the settling of particulate contaminants and is described using charts and tables. These charts assist in determining the obscuration and scatter loss that can be expected when a clean surface is exposed to various Classes of cleanrooms due to particulate settling.

Keywords: cleanliness, aerosols, cleanroom, particulate settling, light scattering, beam obscuration.

1. SCATTER RELATED REASONS FOR ESTABLISHING HIGH CLEANLINESS REQUIREMENTS

The NIF laser system is conceptually divided into small aperture front-end and large aperture high-fluence optics. The smaller front-end optics number over 20,000 and precondition the laser light before entering the 192 symmetric beam-lines.

Because of the large number of serially arranged optical components, it is necessary to achieve and maintain very high levels of surface cleanliness to essentially eliminate all surface scattering losses. Surface cleanliness has been assigned an integrated scatter loss budget of 0.1% for the front-end optics and an additional 0.2% for the large aperture optics. This results in a typical surface scatter loss budget, due to contaminants, of $\leq 2.5 \times 10^{-5}$ per surface. For comparison, the scatter loss per surface due to surface roughness is of a comparable value of 5.0×10^{-5} .

Large aperture optics on NIF have a cleanliness requirement of Level 50-A/10 as installed and will be removed for refinishing if dirt and damage caused obscuration exceeds 2.5×10^{-4} or any single damage site reaches 2-mm in size. The smaller front-end optics must be initially cleaned to Level 100-A and will be removed for refinishing if the accumulated dirt and damage caused obscuration exceeds 2.5×10^{-4} or any single damage site reaches 250-mm in size.

Optical and structural surface cleanliness is further specified as initial cleanliness (immediately after cleaning), as-assembled cleanliness, and end-of-life cleanliness. These cleanliness Levels are defined in Table 1.

2. DAMAGE RELATED REASONS FOR ESTABLISHING HIGH CLEANLINESS REQUIREMENTS

Ample evidence exists that particulate contamination initiates damage on both bare and coated optical surfaces in the presence of high intensity laser light[1]. More recently, it has been found that flashlamp light is sufficient to create aerosols within laser amplifier cavities and that these aerosol particles subsequently settle onto laser amplifier slabs and initiate pitting damage to the optical surface[2]. Stowers, et al, have found that the damage resulting from contaminants can be 7.7 times larger than the contaminant causing the damage.

Table 1 Cleanliness Levels in the as-cleaned, as-assembled, and end-of-life conditions for small optics, large optics, and structural surfaces.

	Surface cleanliness Level as-cleaned	Surface cleanliness Level as-assembled	Surface cleanliness Level at end-of-life
Large optical surfaces	£ Level 50–A/10	Level 50-A/10	£ 1 damage site of 2-mm size per surface or per surface obscuration of 2.5×10^{-4}
Small optical surfaces	£ Level 100-A	Level 100-A	£ 1 damage site of 250 mm size per surface or obscuration of 2.5×10^{-4}
Structural surfaces enclosing large optics	£ Level 83-A/10	Level 100-A/10	Level 100-A/10
Structural surfaces enclosing small optics	£ Level 300-A	Level 300-A	Level 500 (visibly dirty)

It is also now well known that high-intensity laser light is sufficient to dislodge particles from structural surfaces which forms an aerosol that transports particles from structural surfaces to nearby optical surface[3]. If it were not for this laser displacement of contaminants, most contaminants would remain tightly attached to structural surfaces and not transport onto optical surfaces.

The evolution of large lasers has necessitated the need to find more cost-effective optical fabrication processes. One of these is the recent application of thin solgel coating used as anti-reflection coatings on surfaces aligned normal to the laser. Solgel, however, is a material of enormous surface area per gram of coating. It has the unfortunate property that it acts as a getter for organic molecules that may be in inert gases inside laser cavities. The addition of a few monolayers of organic material to the surface of a solgel coating can result in a change in the transmission of the coating of up to several percent. In fact, solgel coated optical surfaces in vacuum (at 10^{-5} Torr) can loose transmission at the rate of up to 0.1% per day in the presence of an inert gas containing a very low concentration of mid-atomic-weight organic matter (organic matter with an atomic weight of 100-200 amu seems to result in the most rapid change in solgel coating transmission). The rapid change in the transmission of solgel coatings in vacuum is exacerbated by large mean-free-path between gas molecules which allows molecules leaving a contaminated surface to be transported nearly ballistically to nearby solgel coated surfaces. At atmospheric pressure this transport mechanism is thwarted by the small mean-free-path of the gas molecules. However, the transport of mid-weight organic molecules still seems to occur at atmospheric pressure, it simply occurs at a substantially lower rate. Solgel coating which remain open and exposed to the air in a Class 100 cleanroom also suffer from the same degradation in transmission and we have repeatedly measured transmission change of 0.1% per month in high quality cleanrooms.

3. SURFACE CLEANLINESS STANDARDS

MIL-STD-1246C *Product Cleanliness Levels and Contamination Control Program*[4] defines surface cleanliness Levels due to particles and thin-films. It has been found that the cumulative size distribution of surface particle contaminants generally follows a \log_{10} concentration versus $(\log_{10} \text{ diameter})^2$ function. A cleanliness Level therefore represents an area concentration of particles exceeding a particular size. Each specific surface cleanliness Level is named for the largest particle size expected to be found on 1 ft² [or 0.1 m²] of surface area. Thus a surface with a Level 100 distribution of contaminants, will have only one (1) particle of 100 mm diameter on each one (1) square foot of surface and an analytically defined number of smaller particles down to 1 mm diameter.

For a particular cleanliness Level (defined by a line in Figure 1) the cumulative concentration is given by the equation below.

$$\frac{\text{particles} \geq \text{diameter}}{\text{ft}^2} = 10^{(0.926 \times (\log_{10}^2(\text{cleanliness Level}) - \log_{10}^2(\text{diameter [mm]})))}$$

For a surface with a cleanliness of Level 100, the concentration of 100 mm and larger particles is found to be 1/ft² and the concentration of 5 mm and larger particles is 1,785 / ft².

MIL-STD 1246C also defines cleanliness Levels associated with thin-film contaminants. Thin-film cleanliness is called Non-Volatile Residue (NVR) and is defined as “material remaining after evaporation of a liquid”. The thin-film cleanliness Level is defined as the mass of the contaminant per ft² [or per 0.1 m²] and is shown in Table 2. In practice, the NVR cleanliness Level is written as an attachment to the end of the particulate cleanliness Level [e.g. Level 100-A/10]. As an indication of the relative cleanliness of Level A/10, a 0.37 nm thick layer of carbon is equivalent to an A/10 cleanliness Level.

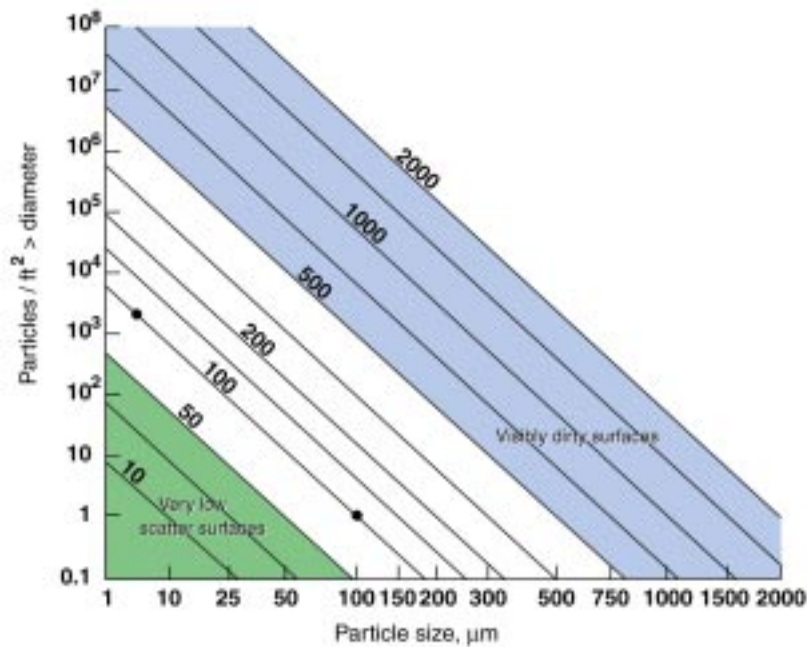


Figure 1 Surface cleanliness chart derived from MIL-STD-1246C. A Level 100 cleanliness Level allows only 1 particles /ft² of 100 mm size or larger and simultaneously allows 1,785 particles /ft² 5 mm size or larger. These two points are shown as small circles on the Level 100 cleanliness line.

Table 2 Thin-film (NVR) cleanliness Levels as defined in MIL-STD 1246C. The A/10 Level is equivalent to a single monolayer of contaminant

NVR Cleanliness Level	Limit, NVR mg/ft ² (or mg/cm ²)
A/100	0.01
A/50	0.02
A/20	0.05
A/10	0.1
A/5	0.2
A/2	0.5
A	1.0
B	2.0
C	3.0

D	4.0
E	5.0

4. SURFACE CLEANLINESS VERIFICATION

Although MIL-STD-1246C defines cleanliness Levels it does not define how to measure it. Measuring the arial concentration of particles on surfaces can be done either *directly* or *indirectly*. Direct examination of very clean surfaces such as Level 100 with $1,785 \text{ particles/ft}^2 = 0.019 \text{ particles/mm}^2 \geq 5 \text{ mm}$ will require the examination of 53 mm^2 at 100x magnification to statistically locate a single 5 mm diameter particle. Since a count of 1 particle after examining 53 areas is not statistically significant, at least 200 mm^2 may need to be examined to achieve a variance of $2 = 4^{1/2}$. In contrast, indirect counting techniques concentrate the particles through liquid flushing or wiping of large areas onto relatively small filter areas followed by counting under a microscope. This mechanical concentration can be expected to result in a 50 to 100x increase in particle concentration and thereby 1) reduce the counting time, 2) improve the counting statistics, and 3) increase the particle concentration so that it is significantly above the background noise of contaminants on the filter paper.

Indirect sampling can be done by flushing a surface with a suitably clean solvent, pouring the contaminated solvent through a membrane filter, and then examining the filter paper under a microscope. If the flushed surface area is significantly larger than the surface area of the filter paper then a relatively large concentration ratio can be achieved. LLNL has developed a filter wiping technique that utilizes a clean dry membrane filter to “swipe” a proscribed area and then the filter paper is examined under a microscope. Unlike the direct examination technique previously described, the swiping distance is adjusted (depending upon the cleanliness Level being verified) to achieve at least 1 particle of 5 mm size in every mm^2 of microscope viewing area. Utilizing very clean filter paper with $\leq 0.1 \text{ particles / mm}^2 \geq 5 \text{ mm}$ and by adjusting the swiping distance to several feet, it is possible to measure particle cleanliness to Level 50. The examination procedure and counting statistics are described in MEL98-012 *Surface Cleanliness Validation by Swiping for NIF Components*[5].

The NVR cleanliness Level can be verified by an indirect sampling process described in MEL98-015 *Measurement of Non-volatile Residue for NIF Components*[6]. The examination process utilizing a very high quality methylene chloride solvent which is used to wash at least 1 ft^2 of surface area and the contaminated run-off fluid is captured in a precleaned bottle. In a cleanroom hood, the fluid containing the NVR residue is concentrated through solvent evaporation and eventually placed on a preweighed cup and weighed on an ultra-microbalance. The weight is divided by the area flushed and reported as mg/ft^2 [or mg/cm^2]. By using good laboratory practices, a background level of 0.02 mg/ft^2 can be achieved. Thus, NVR cleanliness Levels of A/10 can be reliably measured.

5. AIRBORNE CLEANLINESS SPECIFICATIONS

The NIF laser bay, switchyard and target bays are designated Class 10,000. The cleanrooms that perform precision cleaning and assembly are designated as Class 100, and supporting facilities are designated Class 1,000. In contrast, the inside of the laser cavity is designated \leq Class 1.

Airborne cleanliness is designated by “Class” which is a measure of the number of particles/ ft^3 of a size $\geq 0.5 \text{ mm}$ diameter. Details of the metric equivalent classifications can be found in FED-STD-209E *Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zone*[7]. The chart which designates the standard cleanroom Classes is shown in Figure 2. Interestingly, the choice of 0.5 mm for the definition of Class is based on the observation that High Efficiency Particle Air (HEPA) filters tend to have their lowest filtration efficiency at or near 0.5 mm because this is the cross-over point between two different particle capture mechanisms within the filter (diffusion due to Brownian motion dominates the capture mechanism for smaller particles whereas inertial effects dominant for larger particles). These filters actually become more efficient both above and below this cross-over point and designating filter efficiency at this point represent a conservative design philosophy.

6. RELATIONSHIP BETWEEN AIRBORNE CLEANLINESS AND SURFACE CLEANLINESS

Under no circumstances should the designations *Class* and *Level* be used interchangeable. Class refers to the maximum expected particle concentration in a volume of gas whereas Level refers to the maximum particle concentration on a surface. Again, a Class 100 cleanroom is *not* required to achieve or maintain a Level 100 surface, and in fact, maintaining a Level 100 surface is a Class 100 cleanroom is dependent on several variables, the most important being the time of exposure to the air in the cleanroom. The only way to guarantee the maintenance of a Level 100 surface in a cleanroom is to cover it or to place the critical component within a container with an even lower airborne particle concentration or Class.

Otto Hamburg[8] et al have studied the particulate settling rate in operating cleanrooms and found that although there is significant statistical scatter in the data, the rate of surface accumulation is proportional to the airborne concentration, the exposure time, and the orientation of the surface relative to the air flow. Surfaces parallel to the average direction of flow and at right angles to gravity sustain the lowest particle accumulation.

Consider a very simple example; a perfectly clean metallic surface is placed horizontally in a cleanroom designated as Class 100 and which is operating at 100 particles/ft³ ≥ 0.5 mm due to a high population of personnel. The particle settling rate in any room is highly dependent on airflow patterns and electrostatic effects but is dominated by the particle settling velocity (Stoke's velocity) of particles greater than 1 mm in size. The Stoke's velocity V_s of 5.0 mm particles with a density of 1 g/cm³ is 0.12 ft/s. The surface accumulation rate is dependent on the airborne concentration of 5.0 mm particles and the amount of time that the surface remains in the Class 100 environment. The surface accumulation rate is given as the product of the airborne concentration [P/ft³] times the Stoke's velocity [V_s]. Furthermore the total accumulation is the accumulation rate times the exposure time. For our example, the concentration of 5.0 mm particles in a Class 100 cleanroom is not 100/ft³ but can be found by extrapolating the Class 100 line in Figure 2 to the concentration corresponding to the 5.0 mm size particles, which yields a concentration of only 0.63 particles/ft³. Multiplying this by the particles Stoke's velocity of 0.12 ft/min results in an accumulation rate of only 0.076 particles/ft²-min. However, in 24 hours of exposure, the accumulation can be expected to reach 109 particles/ft² ≥ 5.0 mm or a surface cleanliness of Level 44. If the cleanroom had been operating at Class 1,000, then the 24 hour accumulation would be 1,090 particles/ft² ≥ 5.0 mm or a surface cleanliness of Level 87. As shown in Table 1, Level 83 is the desired cleanliness Level of laser cavity surfaces and by simply leaving a "clean" part for 24-hours in a Class 1,000 cleanroom we would expect it to exceed the specified surface cleanliness requirement.

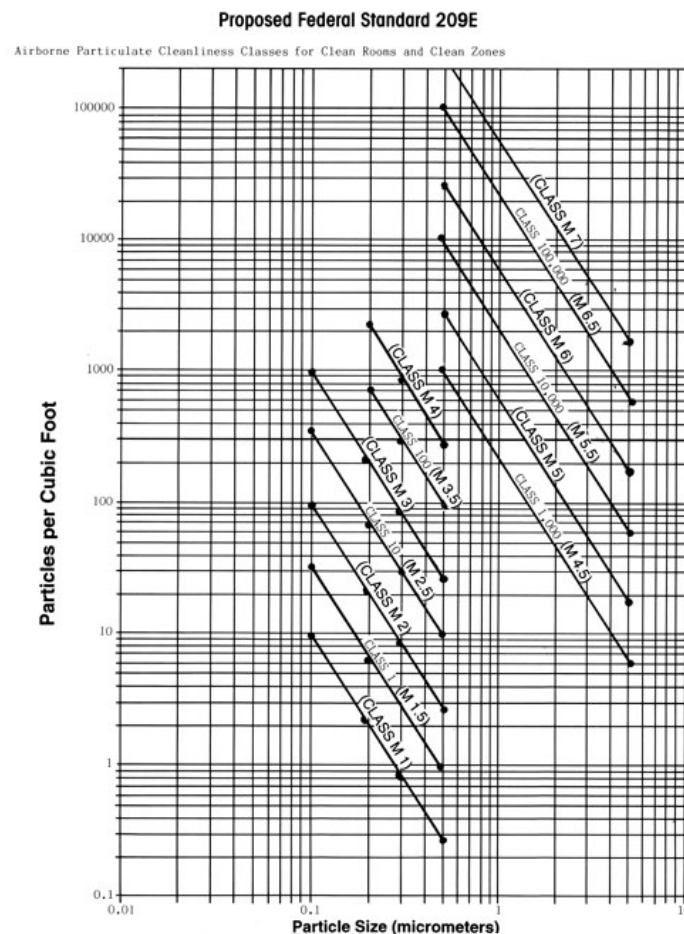


Figure 2 FED-STD 209 defines a series of cumulative size distributions of airborne particles which are named according to where the concentration crosses the particle size of 0.5 mm. A Class 100 cleanroom therefore refers to an airborne concentration of 100 particles/ft³ ≥ 0.5 mm).

7. AIRBORNE CLEANLINESS VERIFICATION

Measuring and verifying airborne cleanliness is easily accomplished using commercially available instruments made possible, in part, by the investment made by the electronics industry and the air pollution industry. Airborne particle counters are available from a large number of vendors and they all work on roughly the same principal; the gas being tested is drawn through a small cell through which a focused light beam passes. The presence of a single particle trips the scattered light sensor which counts the event and the intensity of the light scattered off of the particle determines its equivalent spherical diameter and thereby allows the instrument to assign the counting event to a particular size bin. Current instruments are generally able to measure particles exceeding 0.1 μm in diameter and may report the presence of particles as large as 25 μm . When set to display a concentration of all particles/ ft^3 equal to or exceeding 0.5 mm^3/ft^3 these instruments will directly display the Class of the aerosol in the gas being sampled.

8. CONCLUSIONS

A high degree of surface cleanliness (both particulate and organic) is necessary in laser systems to reduce scattered light and to minimize damage initiated by the presence of particulate matter on optical surfaces. LLNL has established a series of specifications for the surface cleanliness of optical surfaces as well as the structural surfaces surrounding the optics. The Level of particulate and thin-film (NVR) contaminants that are allowed on as-cleaned, and as-installed optics are discussed as well as the level of cleanliness expected at end-of-life. The process for verifying surface cleanliness is discussed and references are given for all pertinent government and LLNL documents. Through experimentation, we have verified that the specified cleanliness Levels are achievable under laboratory conditions and should be achievable under production conditions.

9. ACKNOWLEDGEMENT

The author wishes to thank to following individuals who contributed to the technical content of this paper: Sudhir Jain, Douglas Ravizza, Alan Burnham, John Ertel, Sue Frieders, Thomas McCarville, Dave Camp and James Fair.

10. REFERENCES

- [1] F. Y. Génin, K. Michlitsch, J. Furr, M. R. Kozlowski, and P. Krulevitch, "Laser-induced Damage of Fused Silica at 355 and 1064 nm Initiated by Aluminum Contamination Particles on the Surface", in *Laser-induced Damage in Optical Materials, SPIE Vol. 2966*, 126 (1996).
- [2] I.F. Stowers, J.A. Horvath, J.A. Menapace, A.K. Burnham, and S.A. Letts, "Achieving and Maintaining Cleanliness in NIF Amplifiers", *SPIE Vol. 3492*, 1998.
- [3] Park, H.K.; Grigoropoulos, C.P.; Leung, W.P.; Tam, A.C., "A Practical Excimer Laser-based Cleaning Tool for Removal of Surface Contaminants", *IEEE Transactions on Components, Packaging, and Manufacturing Technology*, Part A, Dec. 1994, vol.17, (no.4):631-43.
- Park, H.K.; Grigoropoulos, C.P.; Yavas, O.; Leung, W.P.; and others, "Efficient Excimer Laser Cleaning for Removal of Surface Contaminants", *Proceedings of 1994 Conference on Lasers and Electro-Optics and The International Electronics Conference CLEO/IQEC*, Anaheim, CA, USA, 8-13 May 1994, Opt. Soc. America, 1994. p. 426-7.
- [4] Institute of Environmental Sciences and Technology, 940 E. Northwest Highway, Mt. Prospect, IL 60056, tel. 847-255-1561.
- [5] Available from LLNL or the author as document NIF5002426.
- [6] Available from LLNL or the author as document NIF5002325.
- [7] Institute of Environmental Sciences and Technology, 940 E. Northwest Highway, Mt. Prospect, IL 60056, tel. 847-255-1561.
- [8] Hamberg, Otto, "Particulate Fallout Predictions for Clean Rooms", *Journal of Environmental Sciences*, Vol. 25, No. 3, May-June 1982, p. 15-20.